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IS 7188 (1974): Methods of test for cellulose acetate flakes [PCD 12: Plastics]



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Indian Standard

METHODS OF TEST FOR
CELLULOSE ACETATE FLAKES

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INDIAN STANDARDS INSTITUTION
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110001

Indian Standard

METHODS OF TEST FOR CELLULOSE ACETATE FLAKES

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Indian Standard

METHODS OF TEST FOR CELLULOSE ACETATE FLAKES

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 10 January 1974, after the draft finalized by the Plastics Sectional Committee had been approved by the Chemical Division Council.

0.2 In the preparation of this standard considerable assistance has been taken from BS 2880:1957 'Methods of testing cellulose acetate flakes' issued by the British Standards Institution.

0.3 In reporting the result of a test made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS:2-1960*.

1. SCOPE

1.1 This standard prescribes the methods of sampling and test for cellulose acetate flakes.

2. TERMINOLOGY

2.1 For the purpose of this standard, the definitions given in IS:2828-1964† shall apply.

3. SAMPLING

3.1 Lot — In any consignment all the material belonging to one batch of manufacture shall be formed into a lot. Each lot shall be tested separately for judging the conformity to the specification.

3.2 Sampling from Bulk — When the supply of cellulose acetate flakes is made in bulk, one lot shall be formed for every 5 tonnes or part thereof. Samples of material from each lot shall be collected at regular intervals when the material is unloaded. The gross sample collected from each lot shall be not less than 5 kg.

*Rules for rounding off numerical values (*revised*).

†Glossary of terms used in plastics industry (*first revision*).

3.3 Sampling from Containers—The number of containers to be selected in the sample from each lot shall be as follows:

<i>Lot Size</i> (<i>Number of Containers</i>)	<i>Sample Size</i>
(<i>N</i>)	(<i>n</i>)
Up to 25	5
26 „ 50	10
51 „ 100	15
101 „ 200	20
201 and above	25

3.3.1 These containers shall be selected at random by using random number tables (*see* IS: 4905-1968*). From each of the selected container equal quality of the material shall be collected and these portions shall be thoroughly mixed to form a composite sample weighing not more than 1 kg.

3.4 Number of Tests and Criteria for Conformity—The gross sample collected under 3.2 or 3.3 shall be packed in airtight containers and shall be used for testing various characteristics. All these tests shall be performed on composite sample unless otherwise agreed to between the purchaser and the supplier.

The lot shall be declared as complying with the requirements of the standard if the test results satisfy the relevant requirements.

4. DETERMINATION ON MOISTURE

4.0 Outline of the Method—The material is heated in an oven at 105 to 110°C for a specified period and loss in mass found out.

4.1 Apparatus

4.1.1 Weighing Bottle—squat type.

4.1.2 Desiccator—containing calcium chloride.

4.2 Procedure—Dry a squat-type glass weighing bottle and stopper in an oven at 105 to 110°C for 30 minutes. Cool in a desiccator over calcium chloride for 30 minutes and weigh accurately when cool. Transfer about 5 g of the sample into the weighing bottle, replace the stopper and weigh again. Dry the material in an oven at 105 to 110°C keeping the lid partially open for a period of 4 hours. Replace the cover of the bottle and cool in a desiccator for 30 minutes. When cool weigh the weighing bottle with contents.

*Methods for random sampling.

4.3 Calculation

$$\text{Moisture, percent by mass} = \frac{(M_2 - M_3)}{(M_2 - M_1)} \times 100$$

where

M_1 = mass of the weighing bottle,

M_2 = mass of the weighing bottle with material before drying,
and

M_3 = mass of the weighing bottle with material after drying.

5. DETERMINATION OF ASH

5.1 Apparatus

5.1.1 *Silica Crucible* — capacity 100 ml.

5.1.2 *Muffle Furnace*

5.2 Procedure—Place a 100 ml silica crucible in a muffle furnace at 500 to 600°C for 15 minutes. Cool the crucible in desiccator for 30 minutes. Find out the mass of the crucible accurately. Weigh accurately about 10 g of the material into the crucible and place over a bunsen flame till the material ignites. Withdraw the flame and allow it to burn. When no further fumes are evolved, apply the bunsen flame again till the bottom of the crucible glows to a dull red. Transfer the crucible to a muffle furnace at 500 to 600°C. When all the carbonaceous matter has burnt, transfer the crucible to a desiccator and cool for 30 minutes and weigh.

5.2.1 Run a moisture determination for the sample simultaneously.

5.3 Calculation

$$\text{Ash, percent by mass} = \frac{(M_3 - M_1)}{(M_2 - M_1)} \times 100 \times \frac{100}{(100 - P)}$$

where

M_1 = mass of empty crucible,

M_2 = mass of crucible with material taken for test,

M_3 = mass of crucible with ash, and

P = percent moisture present in the sample.

6. DETERMINATION OF FREE ACETIC ACID

6.0 Outline of the Method—Extract the free acid by shaking with distilled water. Titrate this against standard sodium hydroxide using bromothymol blue indicator.

6.1 Reagents

6.1.1 Standard Sodium Hydroxide Solution—0.01 N.

6.1.2 Bromothymol Blue Indicator—0.4 percent solution (*m/v*) in water.

6.2 Procedure—Weigh accurately to nearest 1 mg, 6 g of the sample in a 250-ml iodine flask. Add 10 ml of distilled water made neutral to bromothymol blue indicator. Allow to stand for 3 hours with occasional shakings. Filter through Whatman filter paper No. 1 or equivalent. Wash with 50 ml of distilled water neutral to bromothymol blue and catch the washings also. Titrate the filtrate against standard sodium hydroxide to a blue end-point.

NOTE—If the sample contains more than 2 percent by mass of the moisture, dry the sample before weighing.

6.3 Calculation

Free acidity (as CH_3COOH), percent by mass = $V \times N$

where

V = volume in ml of standard sodium hydroxide used in titration, and

N = normality of standard sodium hydroxide solution.

7. DETERMINATION OF ACETIC ACID YIELD

7.0 Outline of the Method—The finely divided cellulose acetate is allowed to stand in contact with a mixture of acetone and sodium hydroxide solution. The amount of alkali consumed for hydrolyzing the material is found out by titration.

7.1 Reagents

7.1.1 Sodium Hydroxide Solution—carbon dioxide-free, approximately N.

7.1.2 Sulphuric Acid—approximately N.

7.1.3 Acetone—95 percent (*m/m*).

7.1.4 Phenolphthalein—6.5 percent solution (*m/v*) in alcohol.

7.2 Procedure—Weigh accurately about 2 g of the finely powdered and dried material into a 250-ml stoppered conical flask. Shake the test portion evenly over the base of the flask and, without lifting the flask from the work-bench, carefully run in 80 ml of acetone and slake for one hour. Add with continual stirring 50 ml of sodium hydroxide and continue shaking for 3 hours. Wash down the stopper with water, adding about 50 ml of water to the contents of the flask. Add exactly 50 ml of sulphuric acid and about 0.5 ml of phenolphthalein indicator. Allow to stand for 30 minutes with occasional shaking. Titrate the excess acid against standard sodium hydroxide. Conduct a blank determination on the reagents used.

7.3 Calculation

$$\text{Acetic acid yield, percent by mass} = \frac{(V_1 - V_2) \times 0.06005 \times N \times 100}{M}$$

where

V_1 = volume in ml of sodium hydroxide solution used in the titration,

V_2 = volume in ml of sodium hydroxide solution used in blank,

N = normality of sodium hydroxide solution, and

M = mass in g of material taken for the test.

8. DETERMINATION OF VISCOSITY

8.0 Outline of the Method—The solution of known strength of the material in acetone is made and viscosity determined using a U-tube viscometer.

8.1 Apparatus

8.1.1 Viscometer—U-tube.

8.2 Reagents

8.2.1 Acetone—95 percent (m/m).

8.3 Procedure—Select an appropriate U-tube viscometer and clean it by rinsing with suitable solvents, followed by ethyl ether. Remove each solvent by passing a current of dry air through it and take care that no moisture remains inside the instrument. Suspend the cleaned viscometer in the bath maintained at $25 \pm 0.5^\circ\text{C}$, taking care that the capillary arm is vertical. Weigh to the nearest 1 mg, 6 g of the dried material into a 250-ml stoppered bottle containing 100 ml of acetone. Stopper the bottle and shake in a linear shaker till the solution is complete. Fill the viscometer with the solution using a long pipette to minimize any wetting of the tube along the filling mark. Allow the viscometer to reach the test temperature and adjust the volume to bring the liquid to within 2 mm of the filling mark. After the sample has reached the test temperature, apply suction or pressure to bring the liquid level up to a point at least 1 cm above the timing mark. Release the suction or pressure and measure the time required for the bottom of the meniscus to pass from the top edges of time marks.

8.4 Calculation

Viscosity at $25 \pm 0.5^\circ\text{C}$, centistokes $= C \times t$

where

C = viscometer constant obtained by calibrating the viscometer using a solution of known viscosity, and

t = time in seconds for the liquid to fall by gravity between the top edges of time marks.

9. DETERMINATION OF CLARITY

9.1 Procedure—Pour a 6 percent (m/v) solution of the material prepared as described in 8.3, slowly through the sides into a clarity tube made of glass, having an outer diameter of 1.4 cm and wall thickness of 1 mm, closed at one end and graduated in centimetres and having a black spot of 4 mm diameter at the flat base end, taking care to avoid air bubbles. Stop pouring the liquid at the point the black spot at the base becomes just invisible when viewed through the liquid column vertically against normal day light. Measure this height of liquid column and report as clarity.

10. DETERMINATION OF HEAT STABILITY

10.1 Procedure—Fill a heat-resistant glass tube 150 mm long, 12.5 mm diameter and 1 mm wall thickness to about 50 mm with the material. Place the test tube in a bath of glycerine or other suitable mineral oil so that half of the column of the sample is above the level of the liquid in the bath. Heat the bath at a uniform rate. When the temperature has reached 180°C , the rate of heating should be regulated to about 2°C per minute. Observe the colour of the sample in the tube. The temperature at which cellulose acetate changes colour to brown is recorded. The heating is continued and the temperature at which the sample fuses is also recorded.

11. DETERMINATION OF AUTOCLAVE STABILITY

11.1 Reagent

11.1.1 Sodium Hydroxide Solution—0.1 N.

11.2 Procedure—Place 6 ± 0.1 g of the sample into a 250-ml flask. Add 100 ml of distilled water and cover with watch-glass. Prepare a blank with 100 ml of distilled water. Place the flask in an autoclave. Heat under a pressure of 1 kgf/cm^2 for 4 hours. Cut off the heating and allow the pressure to come to normal on its own accord. Remove the flask and filter and cellulose acetate over Whatman filter paper No. 41 or equivalent. Wash and titrate the filtrate against standard sodium hydroxide solution (0.1 N) using phenolphthalein as indicator. Carry out a blank using the same amount of distilled water as in the test.

11.3 Calculation

$$\text{Autoclave stability (as CH}_3\text{COOH), percent by mass} = \frac{(V_1 - V_2) \times N \times 0.06005 \times 100}{M}$$

where

V_1 = volume in ml of standard sodium hydroxide used for the sample,

V_2 = volume in ml of standard sodium hydroxide used for blank,

N = normality of standard sodium hydroxide, and

M = mass in g of the sample taken for test.

12. DETERMINATION OF BULK DENSITY

12.1 Apparatus

12.1.1 Funnel—of the following dimensions:

- Vertical height — 98 ± 1 mm
- Diameter of large orifice — 95 ± 1 mm
- Diameter of small orifice — 44 ± 1 mm

12.1.2 Cylindrical Measuring Vessel—of the following dimensions:

- Capacity — 250 ± 1 ml
- Internal diameter — 54 ± 1 mm
- Height — 108 mm approx

12.2 Procedure—Place the funnel in a vertical position and close the small orifice. Place a quantity of the sample exceeding 250 ml in the funnel. Place the measuring vessel with its open end and 25 mm below the coaxial with the funnel. Allow the material to flow into the measuring vessel. When the measuring vessel is full, draw a straight edge across the top to remove excess material. Weigh the contents of the vessel to the nearest 0.1 g.

12.3 Calculation

$$\text{Bulk density, g/ml} = \frac{M}{250}$$

where

M = mass in g of the contents of the vessel.

13. DETERMINATION OF SIEVE ANALYSIS

13.1 Procedure — Carry out the sieve analysis on a suitable portion of the material by dry rate method prescribed in IS:1607-1960* using the following test sieves (see IS:460-1962†):

850 micron, 710 micron, 500 micron, 425 micron, 355 micron,
300 micron, 250 micron, 212 micron, and 150 micron

14. DETERMINATION OF IRON AND COPPER

14.1 Iron

14.1.1 Reagents

14.1.1.1 Hydrochloric acid — 1:1.

14.1.1.2 Ammonium thiocyanate solution — 5 percent (m/v).

14.1.1.3 Standard iron solution — See IS:2316-1968‡.

14.1.2 Procedure — Dissolve the ash obtained in 5 in 10 ml of hydrochloric acid, heat to 60°C and make distinctly ammoniacal and filter. Wash the precipitate free of chloride with ammoniacal water. Collect the filtrate in a 100-ml Nessler cylinder for estimation of copper. Redissolve the precipitate using 10 ml of warm 1:1 hydrochloric acid and transfer into a 100-ml Nessler cylinder. Add 5 ml of ammonium thiocyanate solution and make up to the mark. Match the colour produced against a blank with standard iron solution of known strength.

14.1.3 Calculation

$$\text{Iron (as Fe), ppm} = \frac{V \times 100}{M}$$

where

V = volume in ml of standard iron solution used, and

M = mass in g of the moisture free sample taken.

14.2 Copper

14.2.1 Reagents

14.2.1.1 Sodium diethyldithiocarbamate Solution — 0.1 percent (m/v).

14.2.1.2 Standard copper solution — See IS:2316-1968‡.

*Methods for dry sieving.

†Specification for test sieves (revised).

‡Methods of preparation of standard solutions for colorimetric and volumetric analysis (first revision).

14.2.2 Procedure—To the filtrate obtained from **14.1.2** add 5 ml of sodium diethyldithiocarbamate solution. Match the colour produced against a blank using standard copper solution.

14.2.3 Calculation

$$\text{Copper (as Cu), ppm} = \frac{V \times 100}{M}$$

where

V = volume in ml of standard copper sulphate solution used,
and

M = mass in g of the moisture free sample taken.

INDIAN STANDARDS

ON

THERMOPLASTICS

IS:

- 2267-1972 Polystyrene moulding materials (*first revision*)
- 2530-1963 Methods of test for polyethylene moulding materials and polyethylene compounds
- 2543-1964 Cellulose acetate moulding and extrusion materials
- 3395-1965 Low density polyethylene materials for moulding and extrusion
- 4669-1968 Methods of test for polyvinyl chloride resins
- 5210-1969 High impact polystyrene sheet
- 7166-1974 Cellulose acetate flakes
- 7188-1974 Methods of test for cellulose acetate flakes